Optimizing the Performance of Pilot Vacuum Belt Filter (VBF) for P_2O_5 Reduction of Jordanian Phosphogypsum (PG)

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Inventing new ways to recycle and reuse the accumulated byproducts is the most pressing and daunting challenge facing future process engineers. Millions of tonnes of Phosphogypsum (PG) is stacked in Jordan and worldwide every year. Numerous PG laboratory-scale beneficiation methods are already developed. This research is the first in moving PG Beneficiation methods from laboratory scale to pilot-scale using pilot Vacuum Belt Filter (VBF) to clean PG. In this research, VBF Pilot equipment is designed, constructed, troubleshooted and operated. This pilot study affirmed the difficulty in controlling the process input parameters in pilot VBF when compared with batch filtration. Full factorial (2^3) experimental study is conducted to study the effect of number of washings, number of passes, and acid concentration using sulfuric solutions on PG P_2O_5 content reduction. The three studied parameters showed a significant effect and their interaction was significant and contribute significantly to a considerable reduction in PG P_2O_5 content. The Pilot VBF was successfully operated to achieve an acceptable reduction of PG P_2O_5 content. In this novel pilot VBF research, numerous process insights were practically gained that significantly helped in optimizing VBF performance in reducing P_2O_5 content in PG.

Keywords: Factorial design, Beneficiation, P_2O_5, Phosphogypsum (PG), Vacuum Belt Filter (VBF), Process Optimization.

Introduction

A huge burden of problems is accumulated in the last century that needs to be tackled. Environmental problems take the lead in these problems that humanity faces nowadays and in the future. Solving these environmental problems by inventing new ways to recycle and reuse the accumulated wastes is one of the major challenges for future process engineers. Jordanian fertilizer industry, which is based on local potash and phosphates resources, is one of the major pillars of the Jordanian economy. Jordanian fertilizer industry is mainly based on the production of phosphoric acid. Jordan's annual production of phosphoric acid is estimated to be around 500 thousand metric tonnes (Taib, 2011). In addition to this large amount of phosphoric acid, a five times this quantity is stacked every year as Phosphogypsum (PG) in Aqaba, Jordan. Phosphogypsum (PG) is one of the mineral wastes that is accumulated in large amounts all over the world. The world production of PG is estimated to be 100-280 million tonnes a year that are traditionally stacked in piles. Several impact studies for the stacking of PG show that this practice is uneconomical and adding an ecological burden that needs to be relieved (Reijnders, 2007; Conklin, 1992). Stacks of PG are identified in some 52 countries, including Jordan (Hilton, 2010). PG problem is growing over the years and this PG is in need to be utilized (IFA website). PG has been widely tested and piloted for using it for different purposes, such as plasterboard, plaster, cement and soil additive. Moreover, PG can be used to produce sulfuric acid and the manufacture of ammonium sulphate (IFDC/UNIDO Fertilizer manual, 1998). The commercial use of PG in Jordan is currently limited to the production of cement, as agriculture and soil amendment (Al-Hwaiti *et al*., 2010). PG is progressively considered as an asset more than a waste, but its impurities hinder its recycling. PG accommodates small amounts of numerous mineral impurities that phosphate rock contains, or it is produced in the phosphoric acid production process. Phosphorous and Fluor-containing compounds are the most important group of impurities that are present in PG as P_2O_5 and F respectively (Singh, 2003). Therefore, PG cannot be used for other purposes unless these impurities are reduced to acceptable limits. Reijnders (2007) reported the previously employed methods to reduce the concentrations of minor components in PG. Researchers implemented different methods to clean PG from impurities, such as washing, wet sieving, neutralization with lime, and treatment with a mixture of sulfuric acid and silica or hot aqueous ammonium sulphate solutions (Tayibi, 2009). Many different methods for the reduction of P_2O_5 by chemical and physical methods are already reported in the literature (Saadaouia, *et al*, 2017). Al-Jabbari *et al*., (1988) employed washing PG process with water, sieving it through a 100 μm sieve, and calcining it at different temperatures (low and high). Olmez and Erdem (1989) studied the removal of impurities using several methods based on the neutralization of water-soluble impurities in PG with water and lime milk $Ca(OH)_2$, and a calcining process. Manjit *et al*., (1993) used an aqueous ammonium hydroxide solution (5–20%) to reduce phosphate and fluoride contents in PG. Potgieter *et al*., (2003) studied the effect of chemical and physical treatments of PG used to produce clinker. Klover and Somin (2004) focused their work on the use of a topochemical reaction with an unspecified agent. They claimed that the 226-Ra content of PG can be decreased by a factor of 20–50% and the P_2O_5 content by a factor of 16–28% (Juliastuti, 2018). Aliedeh *et al.*, (2012 and 2018) studied in batch form the dynamic process of leaching of P_2O_5 in PG. In this study, factorial methodologies are designed to study the effect of particle size, acid concentration, loading, and number of washing on the P_2O_5 washing/leaching process using sulfuric and nitric acid solutions. This Multivariate experimental design analysis helped to understand the relative magnitude of the main and the interaction effects. Sulfuric and nitric acid treatment results shed the light clearly on the role of the number of washing on the reduction of P_2O_5 content.

Received on January 22, 2019; accepted on February 25, 2019. Correspondence concerning this article should be addressed to Mohammed Aliedeh (E-mail address: maliedeh@mutah.edu.jo). ORCiD ID of Mohammed Aliedeh https://orcid.org/0000-0003-4458-7846

The optimum conditions for sulfuric acid treatment is found to be offloading 0.15g PG/g solution and three washings. The optimum conditions for nitric acid treatment are found to be of a loading 0.4 g PG/g solution and three washings (Aliedeh, 2012, 2018). Al-Hwaiti (2015a) developed different treatment methods for phosphogypsum: (1) hybrid water, (2) sulphuric acid, (3) mixed acid (H_2SO_4 and HNO_3), (4) household water (tap water and distilled water) and (5) calcium carbonate powder treatment. These treatments are designed to remove and leach the activity concentrations of ^{226}Ra, ^{232}Th, and ^{40}K in the samples of Jordanian phosphogypsum. Al-Hwaiti (2015b) has utilized an inexpensive adsorbent system for the removal of heavy metals from PG using polyethylene glycol and polyvinyl alcohol. This study investigated the performance of these two polymers (PEG and PVA) in removing heavy metal from PG with a batch reaction approach. The above literature review shows that most of the reported research is focusing on lab-scale batch experimental processes without stepping further to pilot scale. Kovler *et al*., (2015) stressed that known PG purification approaches have failed to produce a breakthrough in the industry, and is still confined to the laboratory, largely due to efficiency and cost considerations. Kovler *et al*., (2015) called for cooperation between academic and industrial partners in order to move laboratory-scale approaches to a larger pilot-scale. Aliedeh *et al.*, (2012 and 2018) studied in a batch setup the dynamic process of P_2O_5 reduction in PG through developing a pilot plant model that paved the road to move the PG beneficiation process from Laboratory-Scale to pilot-scale. This research aims to develop and optimize the performance of a pilot-scale VBF process for PG beneficiation and to investigate P_2O_5 reduction using an optimized Pilot Vacuum Belt Filter (VBF) Process.

1 Design methodology: meveloping pilot-scale VBF

1.1 Wet-process of phosphoric acid production

There are two basic types of processes for the production of phosphoric acid; furnace processes and wet processes. Commercial wet processes may be classified according to the hydrate form in which the calcium sulfate crystallizes: Anhydrite -$CaSO_4$, Hemihydrate–$CaSO_4.1/2H_2O$, and Dihydrate- $CaSO_4.2H_2O$ (Singh, 2003). As simple dehydrate process flow sheet is shown in **Figure 1** (Singh, 2003). The role of the filtration and washing process in the wet process aims to separate the produced gypsum from any insoluble materials derived from phosphate rock or formed in the reaction from the phosphoric acid product. All modern plants use only horizontal vacuum belt filters (VBF) (IFDC/UNIDO Fertilizer manual, 1998). The only type of filters that are suitable for phosphoric acid production (Fig. 1) are the horizontal vacuum belt filters (VBFs), which are offered by companies such as Fimco, Delkor, and Pannevis (IFDC/UNIDO Fertilizer manual, 1998).

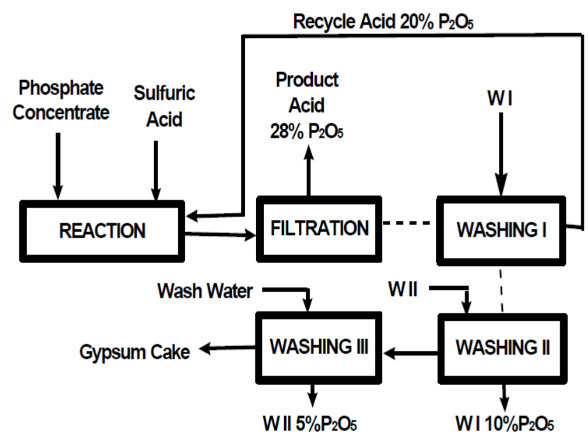

Fig. 1 Simple dihydrate process flowsheet (Singh, 2003)

1.2 Successful applications of vacuum belt filters

Vacuum belt filters (VBFs) can be applied to many different slurries, including fibrous materials, fine slimes, and coarse granular solids. They provide high extraction efficiency, low cake moisture, increased production, and reduced operating costs. VBFs are especially suited for applications requiring low cake moisture and multi-stage washing. Low energy consumption and high filtration rate make VBFs an excellent choice for a wide range of industrial processes (Smidth, 2010). Therefore, VBF is chosen as the equipment to move the PG beneficiation research from the laboratory-scale to the pilot-scale. Moreover, It is implemented in the PG beneficiation research in order to be smoothly integrated into the existing industrial wet phosphoric acid production processes. Horizontal Vacuum Belt Filters are already implemented in almost all wet phosphoric acid plants to separate and wash PG from phosphoric acid solution and its impurities. VBF is characterized by being easily manipulated through controlling slurry feed rate, cake thickness, belt speed, vacuum level, recycling between stages, number of stages, washing rate, racking type, …*etc*. Aliedeh *et al*., 2012 and 2018 did a screen analysis for Jordanian PG and it is found that the PG has a wide spectrum of sizes and it is not, as mentioned, extremely fine. Racking is used in this research to overcome cake resistance. The dynamic manipulation of input parameters in VBF is encouraging implementing it as a tool for PG Abatement.

1.3 Designing the pilot VBF

Based on the established VBF operating guidelines (Smidth, 2010), the Pilot *VBF Process* is designed for three stages: cake formation, cake washing, and cake dewatering, as illustrated in **Figure 2**. The three stages are assembled together to create one pass of a rotating belt filter. Each stage is equipped with a separate filtrate collecting tank that is fitted under the top plate to enable the creation of vacuum in these three trays through connecting them with three separate vacuum pumps, as illustrated in **Figure 3**. The first stage is equipped with a

mixing tank and slurry feed distributor for the PG slurry feed to the cloth and the third stage is equipped with a dry cake scrapping mechanism and a collection container. The dimensions of these numerous Pilot RBF components are shown in **Figure 4**.

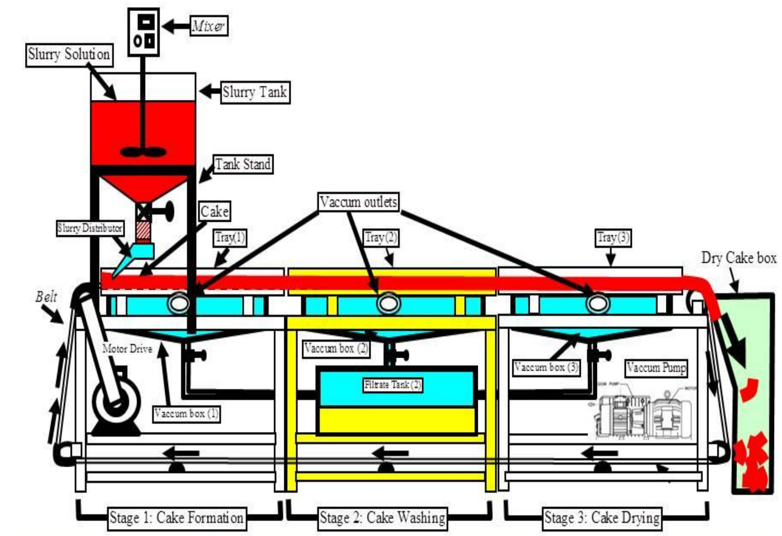

Fig. 2 Main components of the designed Pilot VBF**.**

Fig. 3 Pictures of the designed pilot VBF

1.4 Building and troubleshooting of pilot VBF

The construction, troubleshooting, and operation of this Pilot RBF was a long and daunting process that includes solving the numerous operational challenges and problems such as: slipping of the cloth off the drums and switching to Chaining the rotating cloth; the failure of vacuum build-up and the enhancement of the dewatering process; slurry distributor Problem due to PG solidification; devising an effective mechanism for controlling cake thickness; The selection of an appropriate locally available filter cloth; and proper connection of cloth ends. Even with all these challenges, the researcher achieved the main goal of moving the PG beneficiation process from the laboratory scale to the pilot VBF pilot scale.

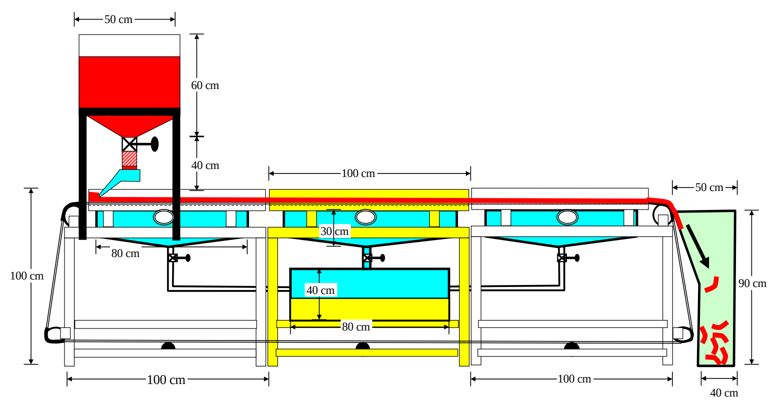

Fig. 4 Dimension of the designed Pilot VBF main components.

2 Experimental methodology: running pilot-scale VBF

2.1 Raw materials

PG samples were collected from Arab Phosphate Co. /Industrial Complex, Aqaba, Jordan. The samples were produced by the dihydrate process. This sample was analyzed for chemical constituents and compared with the composition of PG from other sources as listed in **Table 1**.

Table 1 Chemical compositions of Jordanian PG in comparison to other sources (Aliedeh, *et. al.*, 2012, 2018)

Chemical Compound	PG1*	PG2**	PG3**	PG4**
H_2O	20	19.5	20.0	18.0
SO_2	47.6	43.2	44.0	43.6
CaO	32.60	32.2	31.0	32.0
MgO	0.01	0.01	--	0.40
$Al_2O_3 + Fe_2O_3$	0.173	0.27	0.14	1.82
SiO_2	1.46	1.51	2.40	1.64
Na_2O	0.15	0.47	0.18	0.36
P_2O_5 (total)	1.07	1.01	0.78	1.03
F (total)	0.61	1.67	0.57	0.76
Organic Matter	0.14	0.08	0.24	0.26

* In this study PG1= Arab Phosphate Co., Industrial complex, Aqaba Jordan
** Chandra, S., 1997, PG2=Morocco, PG3=Florida, USA, PG4= India
Chemicals**:** (1) Reagent grade H_2SO_4, (2) Local tap water.

2.2 Determination of phosphates by the gravimetric quimociac technique

Gravimetric Quimociac Technique is based on first converting all the phosphorus-containing species in the sample to soluble orthophosphate (PO_4^{3-}) ion by oxidation and hydrolysis in acid solution. When an acidic quimociac reagent is added to the

prepared orthophosphate sample and a bright yellow precipitate forms. The resulting precipitate is filtered, dried and weighed. Precipitation is described by the following reaction (Shaver, 2008):

$$H_3PO_4 + 12H_2MoO_4 + 3C_9H_7N \rightarrow \underbrace{(C_9H_7N)_3 \cdot H_3PO_4 \cdot 12MoO_3}_{\text{Yellow Precipitate}} \cdot H_2O + 11H_2O \tag{1}$$

When the precipitate is dried, the water of hydration is removed, leaving a stable, anhydrous, yellow product with a molar mass of 2213g/mol.

***Preparation of the Quimociac solution*:**

Dissolve 70g of Sodium molybdate dehydrate ($Na_2MoO_4.2H_2O$) in 150ml distilled water.
Dissolve 60g of Citric Acid Monohydrate ($C_6H_8O_7.H_2O$) in 150ml distilled water, and then add 85ml nitric acid (HNO_3).
Gradually add the molybdate solution to the citric-nitric acid solution while stirring.
Dissolve 5mL synthetic quinoline, with stirring, in a mixture of 35mL of concentrated HNO_3 and 100mL H_2O.
Gradually add this solution to the molybdic-nitric acid solution, mix well and let stand for 24 hours.
Filter, add 280mL of acetone, dilute to 1L with H_2O, and mix.
Store in either a noncolored polyethylene bottle or a dark brown glass bottle.

***Method of Examination*:**

Arrange samples and place them inside porcelain crucibles.
Place the samples in a thermal oven at 105°C, and leave them for 2 hours.
Weight 2.00g of the sample by an analytical balance (±0.0001 accuracy)
Put it in a conical flask.
Add 25ml of digestion solution consisting of:
20% HCl.
40% HNO_3.
40% H_2O.
Put the sample into an electrical heater, until the sample becomes colorless.
Add 50ml of distilled water.
Leave samples for five minutes to get to room temperature.
Put the solution in a volumetric flask, and then add distilled water to the solution until fully filled (250 ml).
Recap volumetric flask tightly and then shake the solution very well.
Filter the solution by filter paper.
Draw 50 ml (aliquot) of filtered sample by pipette, and place it in a beaker.
Add 25 ml of nitric acid solution consisting of:
33.33% HNO_3.
66.66% H_2O.
Add 25ml of Quimociac solution.
Place the solution on an electric heater until it becomes a dark yellow color.
Leave samples for five minutes to get to room temperature.
Weight the crucibles.
Filter sample by vacuum pump filter.
Put the crucibles in a thermal oven at 105°C, and leave for 2.5 hours.
Calculate the weight of the precipitate
Calculate $\%P_2O_5$ based on the following equation:

$$\text{Percent } P_2O_5 = \frac{\overbrace{\overbrace{\underbrace{\underbrace{\frac{(\text{g precipitate})}{\left(2213\,\frac{\text{g precipitate}}{\text{mol precipitate}}\right)}}_{\text{mol precipitate}}\left(\frac{\text{mol } P_2O_5}{2\text{ mol precipitate}}\right)}_{\text{mol } P_2O_5}\left(141.9\,\frac{\text{g } P_2O_5}{\text{mol } P_2O_5}\right)}^{\text{g } P_2O_5 \text{ in sample aliquot}}\left(\begin{array}{l}\text{Sample}\\ \text{Volme Ratio}\end{array}\right)}^{\text{g } P_2O_5 \text{ in sample solution}}}{(\text{g sample})}(100\%) \tag{2}$$

This can be arranged to result in:

$$\text{Percent } P_2O_5 = \frac{(\text{g precipitate})\left(141.9\,\frac{\text{g } P_2O_5}{\text{mol } P_2O_5}\right)\left(\begin{array}{l}\text{Sample}\\ \text{Volme Ratio}\end{array}\right)(100\%)}{\left(2213\,\frac{\text{g precipitate}}{\text{mol precipitate}}\right)\left(2\,\frac{\text{mol precipitate}}{\text{mol } P_2O_5}\right)(\text{g sample})} \tag{3}$$

$$\left(\begin{array}{l}\text{Sample}\\ \text{Volme Ratio}\end{array}\right) = \frac{\text{volume sample solution}}{\text{volume sample aliquot}} = \frac{250\text{ ml}}{50\text{ ml}} \tag{4}$$

2.3 Factorial design methodology

Experimental designs are plans for determining how one or more experiments are to be run. Experimental strategies range from trial-and-error testing through one- factor or variable-at-a-time testing (OVAAT) to what is more commonly considered Design of Experiments (DOE) such as factorial designs (Smidth, 2010, Moalla, 2018). Full factorial design (23) is implemented in this research because it allows measurement of the main effects in addition to the interaction effects needed to obtain high levels of complex process understanding. The full factorial design of 23 is implemented with three input parameters that are selected to be studied: (1) Number of washing, (2) number of Passes, and the concentration of aqueous solution, as listed in **Table 2**. Two replicates for each run is accomplished in addition to six center points as shown in the design matrix listed in **Table 3**.

Table 2 The selected values of input parameters in the full factorial experimental design matrix

Input Parameters	Low	Center	High
# of Washing (A)	1	2	3
# of passes (B)	1	2	3
% H_2SO_4 (w/w) (C)	1	3	5

Table 3 Experimental design matrix and results for P_2O_5 wt% in pilot VBF treated PG samples using sulfuric acid solution

Run	No. of Washes	No. of Passes	% H_2SO_4	No. of Washes	No. of Passes	% H_2SO_4	% P2O5
1	-1	-1	-1	1	1	1	0.7648
2	+1	-1	-1	3	1	1	0.6642
3	-1	+1	-1	1	3	1	0.5708
4	+1	+1	-1	3	3	1	0.736
5	-1	-1	+1	1	1	5	0.7551
6	+1	-1	+1	3	1	5	0.6231
7	-1	+1	+1	1	3	5	0.5432
8	+1	+1	+1	3	3	5	0.3384
9	-1	-1	-1	1	1	1	0.828
10	+1	-1	-1	3	1	1	0.6883
11	-1	+1	-1	1	3	1	0.6991
12	+1	+1	-1	3	3	1	0.792
13	-1	-1	+1	1	1	5	0.7654
14	+1	-1	+1	3	1	5	0.7221
15	-1	+1	+1	1	3	5	0.5852
16	+1	+1	+1	3	3	5	0.3665
17	0	0	0	2	2	3	0.5048
18	0	0	0	2	2	3	0.5576
19	0	0	0	2	2	3	0.5288
20	0	0	0	2	2	3	0.5767
21	0	0	0	2	2	3	0.5279
22	0	0	0	2	2	3	0.5463

2.4 VBF Pilot plant operating conditions

The PG samples were thoroughly mixed with aqueous sulphuric Acid solution in the feed tank for 30 minutes. The PG/ Sulfuric Acid Solution mixture is processed through the VBF pilot equipment based on the operating conditions listed in **Table 4**. The control of these important process parameters was an added challenge to the experimental work accomplished. Some variation is noticed in some of these experimental operating conditions such as washing rate, film thickness, Solution ration due to settling and feed rate. These sources of variability are widening the range of variation in the P_2O_5 reduction achieved.

3 Results and Discussion

Full ANOVA analysis is applied to the 2^3 full factorial design of the pilot study (The Design matrix is shown in Table 3) using Minitab® 16 software, as listed in **Table 5**. The ANOVA analysis results are plotted in the form of Pareto chart (**Figure 5**) to show the significance of each studied input variable effects.

3.1 Main effects

The significance of input variables is determined based on the value of probability p. Full ANOVA analysis shows that the number of washes (A), the number of passes (B) and Acid Concentration (C) were significant within the 95% confidence interval ($p \leq 0.05$). These three input variables are expected to play an important role in reducing the PG P_2O_5 content (Fig. 5 and **Figure 6**). These results agree well with the previously published batch dynamic results (Aliedeh, 2012 and 2018), i.e. the number of washes, number of passes and higher acid concentration significantly contribute to more reduction in PG P_2O_5 content. This indicates that the pilot VBF is functioning properly and it can be a good basis to move towards pilot plant scale.

Table 4 VBF pilot equipment based on the operating conditions

Operating Conditions	Condition Value
PG/ Solution Ratio	0.5 g PG / g Solution
PG mixture feed rate to VBF	0.25 kg PG mixture/minute
Film thickness	3-5 mm
Washing rate	0.5 liter/minute
Filtration Belt Moving Speed	1.5 m/ minute
Cloth Material	Polyester (PET)

3.2 Interaction effects

This study is based on the multivariate experimental work; therefore, interaction effects must be discussed with the main effects. Factorial design methodology can reveal the interaction between the input parameters under study. The focus of this study will be on the two-factor interactions. **Figure 7** shows the two-factor interaction plots for the study of VBF P_2O_5 reduction by sulphuric acid solution. These plots show that all the interactions are almost significant. Strong interaction is observed between the number of passes (B) and the acid solution concentration (C), the number of washes (A) and the acid solution concentration (C), but a weak interaction is clear between the number of washes (A) and number of passes (B)

Table 5 Full 2^3 factorial design for P_2O_5 wt% in VBF treated PG samples using sulfuric acid solution.

Term	Effect	Coef	SE Coef	T	P
Constant		0.6526	0.01017	64.16	0
A	-0.0726	-0.0363	0.01017	-3.57	0.003
B	-0.1475	-0.0737	0.01017	-7.25	0
C	-0.1305	-0.0653	0.01017	-6.42	0
A*B	0.0313	0.0156	0.01017	1.54	0.148
A*C	-0.0771	-0.0385	0.01017	-3.79	0.002
B*C	-0.1106	-0.0553	0.01017	-5.44	0
A*B*C	-0.0933	-0.0467	0.01017	-4.59	0.001
Ct Pt		-0.1123	0.01948	-5.76	0

Coef= Regression Coefficient , SE Coef= Standard Error of Regression Coefficient, T=Student's Distribution Value, P=Probability Value for t Distribution

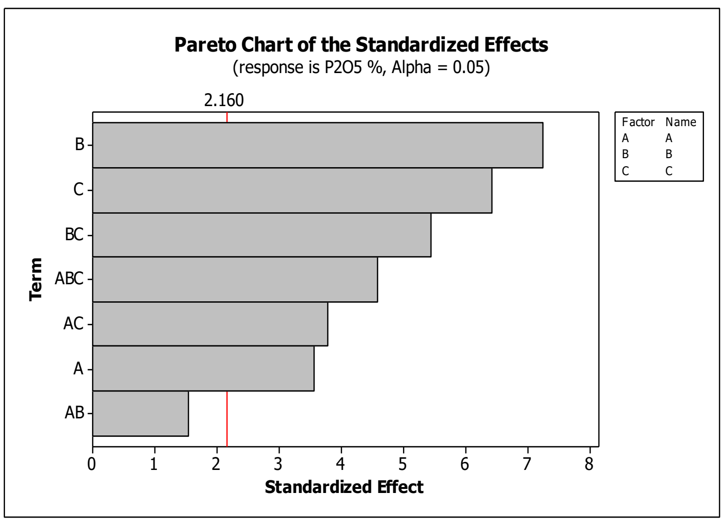

Fig. 5 Puerto chart for sulfuric acid treatment 2^3 full factorial design experiments.

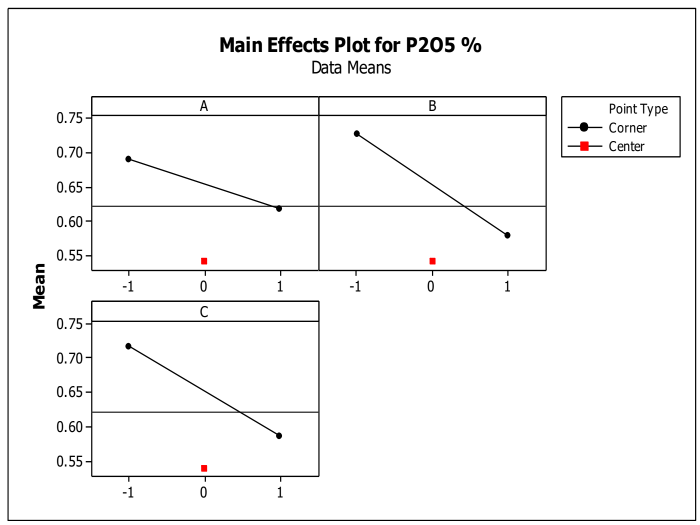

Fig. 6 Main effect plots for sulfuric acid treatment 2^3 full factorial design experiment

This interaction behavior totally agrees with the interaction pattern that was reported in Aliedeh *et al.*, (2012 and 2018) batch dynamic studies. These strong interaction effects can contribute to affect a change in P_2O_5 reduction behavior. The results show the selection of input parameters in both batch dynamic studies (Aliedeh, 2012 and 2018) and the pilot VBF study was successful and both agree on the most important parameters that the P_2O_5 reduction process should focus on (number of washing, number of passes, and acid concentration). These three important process parameters and their interactions are integrated to get a considerable reduction in P_2O_5 content. Based on the above discussion of the results, it should be noted that the sources of errors and interference in pilot-scale experiments are significantly more pronounced than in batch-wise experiments.

The control of errors in continuous pilot plant studies is very difficult and requires long experience in running these pilot plants effectively. Despite these challenges, both the previous batch dynamic studies and this pilot VBF research successfully achieved an acceptable amount of P_2O_5 content reduction and clearly identified the most important input parameters that strongly contribute to the PG P_2O_5 content reduction process. The strong effect of the number of washings stressed the importance of maintaining an effective washing process by: (1) controlling effectively the PG slurry thickness (2) minimizing channeling during cake washing over the filter cloth by slice fragmentation, as illustrated in **Figure 8**. These cake washing problems did not show up in dynamic batch results experiment (Aliedeh, *et al.*, 2012 and 2018), because a Buchner funnel filtration was used in which a film thickness of 2-3 mm and a highly controlled washing is affected by spraying water all over the Buchner funnel and manually plowing the PG cake formed, see **Figure 9**. It is recommended that more attention should be given to a new pattern of plowing the cake in order to enhance its washing process over the rotating filter cloth, as illustrated in Fig. Further current research in our labs is being accomplished to handle the above-mentioned problems.

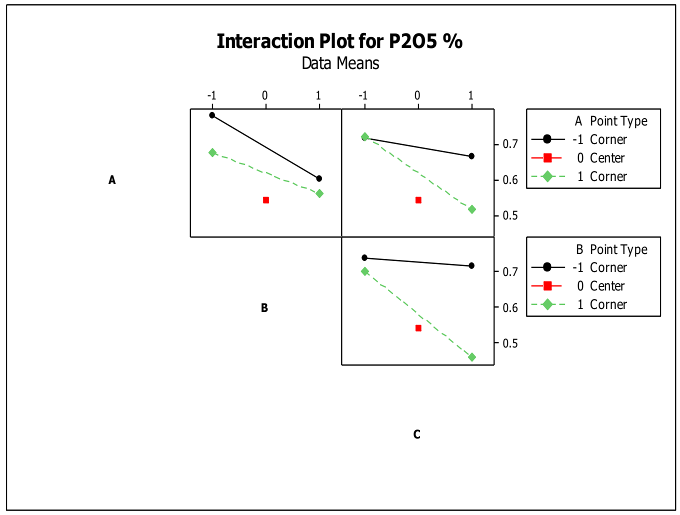

Fig. 7 Interaction effects for sulfuric acid treatment 2^4 full factorial design experiment

Fig. 8 Photographs of racking patterns of the PG cake while moving over the rotation belt

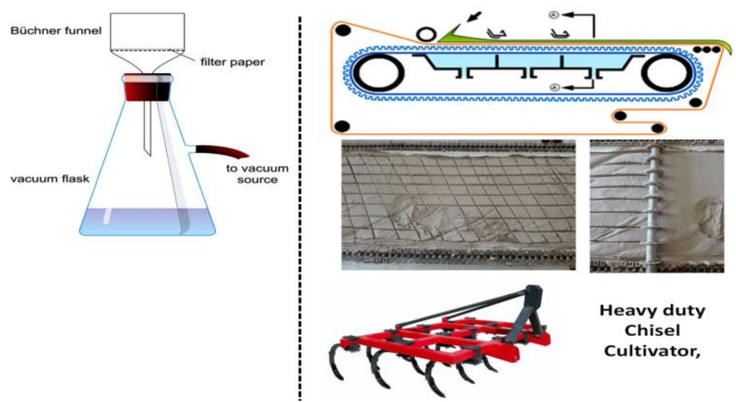

Fig. 9 The need to plough the filtered cake to enhance the washing over the belt

Conclusions

The added value of this research is manifested in by achieving two shifts in PG conventional research; shifting from lab scale to the pilot plant scale, and shifting from one variable at a time (OVAAT) to factorial design research methodology. The courageous attempt to shift our PG conventional research from lab scale to the pilot plant scale was the most challenging research approach shift. Pilot scale research is considered the connecting ring between the lab scale and industrial scale. The highest and the most challenging job lie in the pilot plant scale because it is characterized by being daunting, time consuming, and challenging type of research that encompasses basic research and engineering research methodologies. The challenges lie in the construction and running the pilot plant to make it work appropriately in the continuous mode. Feeding the PG slurry, uniformity of PG cake on the cloth, finding the proper filter cloth, failure of vacuum on trays, scrapping the cake off the cloth, slippage of cloth from rotating drums are some of long list of daunting operating problems. In addition, it should be noted that the sources of errors and interference in pilot scale experiments are significantly more pronounced than in batch wise experiments. The control of errors in continuous pilot plant studies is very difficult and requires long experience in running these pilot plants effectively. In spite of these challenges, this research was successful in taking PG research a step further to the pilot process research with acceptable research results. The shift from OVAAT to Factorial Design research (frequently called Design of Experiments, DOE)

methodologies is another added value in this research work. As shown in the collected DOE research results, the focus is not only on the main effects, but it also gives an equal emphasis is given to the interaction between the studied parameters (Main and Interaction Effects).

Acknowledgments

This work was supported by The Jordanian Scientific Research Support Fund (SRRF) [Grant number: Eng/2/12/2013]. The author acknowledges Eng. Atif Altarawneh for laboratory support.

Nomenclature

g precipitate	=mass of precipitate	[g]
Percent P_2O_5	=mass percent of P_2O_5	[m]
g sample	=mass of PG solid samples used for analysis	[g]

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